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FINAL PROGRESS REPORT

JPL Contract No. 952091
Development & Fabrication of
Improved Positive Expulsion Bladders

Submitted to:

California Institute of Technology
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Pasadena, California

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NAS 7-100

The exercise of this contract was undertaken in two Phases. Phase I was subcontracted to the Commonwealth Scientific Corp. of 500 Pendleton Street, Alexandria, Virginia, under Dilectrix subcontract No. 8271. Phase II was accomplished at the Dilectrix Corporation facilities at Farmingdale, New York.

Physical specimens of Teflon/aluminum composites were prepared and a number of these specimens have been submitted to JPL as inclusion in Interim Reports Nos. 1 through 7. Additional specimens are being submitted with the final report as Exhibits attached hereto.

The correlation between the specimen Exhibits and the reports themselves follows in summary:

Exhibit	
1 through 4 and 7	See Appendix (Table of Contents Phase II) *
5 and 6	Dilectrix prepared specimens, Phase II *
8	Dilectrix prepared specimens, reference Table XI, pg. II-32A through II-32D, Phase II. *
9	Commonwealth prepared specimens, reference Table 1, pg. 8, Phase I. **
10	Commonwealth prepared specimens, reference Tables VI and VII, pgs. 49-51, Phase I. **

* FINAL PROGRESS REPORT DATED 27 August 1968

** FINAL REPORT 31 July 1968

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A. DISCLOSURE

This report has been prepared in compliance with JPL contract No. 952091 documentation requirements for the purpose of disclosing to Jet Propulsion Laboratory the progress and results of the effort accomplished at Dilectrix during the contract.

I. INTRODUCTION

A. Past History of Laminating Teflon.

Dilectrix has manufactured and supplied Teflon positive expulsion bladders for space and military applications for nearly ten (10) years. During this time it has continually investigated all possible avenues tending to improve reliability of these devices. Increased shelf life, increased flexural capability and reduced permeability have undergone continuous scrutinization in R & D programs since 1960.

The very first steps of this work were directed toward studies of basic properties of dispersion cast films and spray coatings employing commercially available "Teflon" fluorocarbon resins. As a result of these studies Dilectrix has now available numerous formulations based on the two classes of Teflon "TFE" and "FEP" resins. The fundamental studies on the crystallinity and molecular arrangements of these resins led to the development of adapting a laminate construction of the two materials and later to the more advantageous system of using the two in co-dispersion form in proportions to obtain minimal permeation and satisfactory flexural performance. Continued studies on permeation failures and stress defects led to a variety of bladder constructions including triple ply laminates, redundant laminations and finally to metal barrier laminates. With each configuration bladder efficiency increased. Other systems were then explored to improve certain properties while simultaneously maintaining quality gains previously achieved in other properties.

B. Reason for Initiating the Present Program.

Since maximum expulsion efficiency is a basic requirement for a flexible propellent container the materials used for this purpose must necessarily respond to high flexibility demands for repetitive expulsion cycles without impairing the integrity of the membrane. Further, the materials employed in the construction of these units must be compatible with propellent fuels and oxidizers. Many fluorocarbon resins and certain selected elastomers comply with the above requirements.

Of the above, Dilectrix has found TFE to be the most acceptable bladder material. However, the presence of micro voids in the finished film, inherent to the high crystallinity of this fluorocarbon, makes it highly permeable to aerospace fuels and oxidizers.

Therefore, Dilectrix has conducted an extensive in-house research program for the development of bladder wall barriers, and has taken a multiple approach to minimizing the permeation problem.

Chemical, vacuum, electro-dip and electro-brush plating of the films, inclusive of metal flake barriers, impregnation of the film with catalyzed siloxane oils and lamination of metallic foil barriers, among others, have been tried, singularly and in combination, in an attempt to solve the permeation problem.

It has been determined that micro sized metallic flakes interposed between TFE bladder laminations reduced N_2O_4 permeation over 50%. However, flex life was also substantially increased with metallic flake intra-laminates.

Results with metal barriers applied by vacuum deposition of aluminum onto the base Teflon showed that layers heavier than 20-25 micro inches would flake and crack, while thinner layers did not prevent permeation. Additional efforts in chemical and electrical plating over sensitized base Teflon did not provide the necessary flexibility.

Lamination of a solid rolled aluminum foil between Teflon film layers gave the most promise of producing a bladder with near zero permeability while retaining the physical integrity of the Teflon. An intensification of this investigation was warranted so that a definite and conclusive solution to the permeation problem could be ascertained.

C. Reason For Working Jointly With Commonwealth Scientific Corp.

In addition to rolled aluminium foil, a technique pioneered and developed by the personnel of Commonwealth Scientific Corp., Alexandria, Virginia, of chemically vapor depositing aluminum on Teflon substrates to obtain a seamless permeation barrier, was investigated. Aware of the promise offered by a bladder wall construction with contiguous metallic barrier lamination, Dilectrix allied its production and technical capabilities with the scientific and technical capabilities of Commonwealth Scientific.

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II. ABSTRACT.

A. Contract Goals.

The goals of Phase II of JPL Contract 952091 were as follows:

1. Investigation of Metal Foil Laminates.

   A. Improve techniques for bonding metals to Teflon.

   B. Optimize arrangement of materials for improved flexibility.

   C. Find methods of applying metal barrier to completed bladders.

2. Physical Property Goals.

   A. Reduced permeability of propellants and propellant gases.

   B. Increased cycle life.

   C. Freedom from delamination of the constituent parts.

3. Metallic Foil Investigation.

   A. Aluminum foil investigation for optimum type and preparation.

   B. Stainless steel foil investigation for optimum type and preparation.

B. Contract Accomplishments.

1. Optimization of the foil to be used in the program to 1/2 mil aluminum foil.

2. Optimization of Autoclave techniques and arrangement of material in the laminate so that the surface appearance can be predicted and blister free laminates can be repeatedly produced.

3. Employment of co-dispersions of TFE and FEP in varying proportions to improve physical properties of the laminate.

4. Elimination of any propellant or gas permeation by the inclusion of the foil.

5. Attainment of flexural cycle life which is acceptable when the laminate is rolled upon itself. Foil pinholding does occur, however, when the JPL flex tester is employed.

6. A tensile strength at yield of the laminates in the 2500-2900 psi range.

7. A percent elongation at yield of 23-41%.

8. An initial modulus of about 300 kpsi.

9. A bond strength before 96 hour N_2O_4 soak of 3.5#/in./in.

10. All work initially outlined in the contract has been accomplished to the extent of the contract. Samples from Phase II and all of Phase I samples are being included with the final report.

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III. Task 1 - Metallic Film Studies.

A. Aluminum Foil Studies.

(1) Selection of Samples.

Aluminum foils were investigated to determine their availability, cost, maximum purity, ductility, pinhole frequency and cleaning and surface treatment. It was determined that many suppliers could deliver aluminum foil in 99.45% purity which constitutes a high grade electrical capacitor foil, in 1/4, 1/2, 3/4 and 1 mil thicknesses. One mil foil of 99.99% purity was also found to be available, however, the extreme softness due to its high purity invariably resulted in a high frequency of pinhole, therefore this particular grade of foil was not employed.

All of the aluminum foils selected for study were purchased from Republic Foil Corporation, Danbury, Connecticut. These foils were classified as being: "One Side Bright", "Driwind" or "Electrodry", referring to a cleanliness level as determined by the manufacturer. All grades were heat cleaned and annealed prior to use and were presumed to be free from any organic surface contaminants.

(2) Pinhole Frequency Study.

The aluminum foils chosen were examined for determination of pinhole size and frequency as related to foil thickness. The method used for this operation was simply a photographic light box. Each sample was secured against the frosted glass surface of the internally illuminated box, located in a dark room. When a 100 watt inner lamp was energized pinholes in the samples were clearly visible and the size randomly measured with a comparator (Edmund Scientific Co.).

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All foil samples were identified by a number and pinhole count information recorded for each. At the conclusion of this test the results were summarized and tabulated in Appendix I, Table I. The size and frequency of inherent pinholes reduce measurably as foil thickness increases.

The following pinhole photographs were taken of three foil samples and are included in the appendix as Exhibits 1, 2 and 3:

1 photo of .00025 aluminum foil, roll 1, of case 16895.

1 photo of .00025 aluminum foil, roll 2, of case 16895.

1 photo of .0005 aluminum foil, from case 781.

These were prepared as outlined below:

(1) Standard commercial photographic and dark room equipment and materials were used for this test.

(2) A 12" x 12" opaque glass mask was prepared with a 6-1/2" x 8-1/2" clear area wherein the foil was exposed against photographic paper.

(3) Aluminum foil layed over clear area of the glass mask with glossy side over glass.

(4) Photographic paper placed over foil with sensitized side over the aluminum.

(5) Exposure of paper negative against light for a one minute period.

(6) Development drying and trimming.

Table I

Aluminum Foil Pinhole Studies

	Source	Case No.	Roll No.	Thickness	Sample Size	Number and Size Pinholes < .005	> .005	> .010	> .020
1	Republic Foil Corp.	16895	1	.00025	4" x 4"	112	28	2	1
2	" " "	16895	1	.00025	4" x 4"	157	12	4	0
3	" " "	16895	1	.00025	4" x 4"	201	27	1	1
4	" " "	16895	2	.00025	4" x 4"	312	34	1	0
5	" " "	16895	2	.00025	4" x 4"	246	22	2	0
6	Republic Foil Corp.	17675	1	.0005	1' x 1'	0	0	0	0
7	" " "	17675	1	.0005	1' x 1'	1	1	0	0
8	" " "	17675	1	.0005	1' x 1'	1	0	0	0
9	" " "	17675	1	.0005	1' x 1'	0	0	0	0
10	" " "	17675	1	.0005	1' x 1'	0	0	0	0
11	" " "	17675	1	.0005	1' x 1'	0	0	0	0
12	" " "	17675	1	.0005	1' x 1'	1	0	0	0
13*	Republic Foil Corp.	781	1	.0005	1' x 2'	25	6	7	0
14	" " "	781	1	.0005	1' x 2'	0	0	0	0
15	" " "	781	1	.0005	1' x 2'	1	0	0	0
16	Republic Foil Corp.	40135	1	.0007	1' x 1'	0	0	0	0
17	" " "	40135	1	.0007	1' x 1'	0	0	0	0
18	" " "	40135	1	.0007	1' x 1'	0	0	0	0
19	Republic Foil Corp.	27241	1	.001	1' x 15'	0	0	0	0

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*Pinholes in sample #13 were confined to an area 3/4" in diameter.

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It should be noted that handling during lay-up, even with the exercise of extreme care, results in small creases which cause porosity and possibly other faults in the foil. The presence of these few pinholes is not a serious problem, as these voids are mitigated by the flow of Teflon at fusing temperature and by overlapping of foil. Pinholes were induced in several sections which were later coated with Teflon, permeation tests produced negative results illustrating that low frequency does not influence permeation.

(3) Heat Treatment of Foil Samples.

A further investigation performed consisted of subjecting aluminum foil samples to heat treatment to determine the feasibility of increasing their ductility. Physical constant tests were run on heat treated and non-heat treated sections of the following:

.00025 thick "Electrodry" Case No. 16895, roll 1

.0005 thick "Electrodry" Case No. 17675

.0007 thick "Driwind" Case No. 40135

.001 thick "Electrodry" Case No. 27241

Method of Aluminum Heat Treatment.

(1) Aluminum foil samples were placed inside a protective metal envelope to prevent them from floating or being otherwise damaged inside of the vacuum retort used in heat treating.

(2) The metal envelope was placed on a sheet of asbestos inside the vacuum retort.

(3) A vacuum line and an argon gas line, with fittings to connect a flow meter and mecury manometer, were connected to the retort.

(4) With the argon line closed a vacuum of 500 mm. of mercury was drawn on the retort. After reaching 500 mm. the vacuum line was shut and the retort was purged with argon gas.

(5) Purging with argon as described in Step 4 was repeated twice.

(6) With the vacuum line closed the flow meter was set on the argon gas cylinder to deliver 20 cubic feet of argon per hour.

(7) A thermocouple was connected through an entrance port on top of the retort.

(8) The retort was placed in a preheated gas oven at 800°F.

(9) Recorded temperatures and time until T.C. reached 775° After 1.5 hours at a minimum of 775°F the oven was slowly cooled to approximately 200°F.

The total heating and cooling operation schedule was as follows:

Operation	Time
Assembly placed in oven	0
Attain max. temperature, 775°F	.5 hr.
Held at max. temperature	1.5 hr.
Total time to cool (190°F)	2.0 hr.

(10) Turned off argon and removed samples from vacuum chamber.

(11) Sent heat treated samples to testing laboratory along with untreated samples for physical tests.

(12) Results of aluminum foil physical tests are shown in Table II. All values are averages of six determinations on each foil sample. The tests were performed on an Instron Tensilometer at an

outside physical test laboratory and showed that except for 1/4 mil (.00025 in.) foil the heat treatment does not provide a significant reduction in modulus, consequently there appears to be no advantage to be gained with regard to higher degree of ductility.

(4) Cleaning of Foil Samples.

An investigation was made into various methods of cleaning aluminum foils. Etching and solvent cleaning systems were explored using metal foils in the "as received" condition.

Etching was accomplished by means of immersing samples of foil in various concentrations of sodium hydroxide solution which had been sequestered with sodium gluconate. Concentrations of 50%, 25%, 12% and 7% NaOH were made up and aluminum foil samples of various thicknesses immersed for various time periods. Immediate problems encountered were quick reaction on thin foils with subsequent dissolution and/or massive pinholing of the foil and difficulty in rinsing the tenacious caustic solution from the foil surfaces without wrinkling or otherwise damaging the thin metal.

Solvent cleaning was accomplished by means of immersing samples of foil in acetone (reagent grade) for a period of 1/2 hour and then placing the samples in a warm air recirculating oven.

The water droplet test was used to compare the "cleaned" foils with the "as received" foils. This test involves three drops of deionized water dripping on a singular spot on the metal foil, from a fixed height of one inch. The area of water spread was then measured diametrically. This method is akin to the popular water "break" test, however, for thin foils the water drop test is more convenient.

Table II

Physical Test Results on Task 1 Foils

Sample	Thickness*	Modulus K PSI*	Elongation % *	Tensile Break K PSI*
Al. .00025" Not heat treated	0.15	7,067,000	10.5	22,226
Al. 00025" Heat treated	0.15	4,796,000	21.3	15,818
Al. 0005" Not heat treated	0.53	2,525,000	20.4	6,910
Al. 0005" Heat Treated	0.53	2,782,000	23.0	6,791
Al. 0007" Not Heat Treated	0.71	2,521,000	39.5	9,201
Al. 0007" Heat treated	0.72	2,525,000	36.3	7,629
Al. 001" Not heat treated	1.00	3,453,000	27.4	7,487
Al. 001" Heat treated	0.98	3,158,000	28.8	6,809
.0001" S.S. Type 304 Not heat treated	0.13	13,857,000	10.4	146,910
.0005" S.S. Type 304 Not heat treated	0.55	13,917,000	25.9	237,400
.00078" S.S. Type 304 Not heat treated	0.80	12,300,000	25.7	223,370

* All values are from an average of 6 samples tested.

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Careful measurement of water spread on "cleaned" versus "as received" foil surfaces failed to indicate a significant increase in wettability in the cleaned foils.

B. Stainless Steel Foil Studies.

(1) Selection of Samples.

An investigation was also made to determine availability, cost, ductility, possible heat treatments, pinhole frequency and surface treatments of stainless steel foils.

Stainless steel foils are obtainable in grades 301, 304, 305 with grade 302 made on special request in thicknesses from 1/10 to 1 mil. Types 304 and 305 are the most malleable foils as they contain lower concentrations of carbon, with 304 containing the lowest carbon.

Samples chosen for this program were obtained from Hamilton Watch Co. of Lancaster, Penn. They were of the type 304 in a cold rolled condition. Samples obtained were in sheet sizes, .0001 x 4 1/4" x 5', .0005 x 4-1/4" x 5' and .00078 x 8-1/4" x 5' .

(2) Pinhole Frequency Study.

Each sample of stainless steel foil was subjected to pinhole frequency studies using the same method described for pinhole counts on aluminum foils. No pinholes were found in any of the samples.

(3) Heat Treatment of Stainless Steel Foils.

A study was also made into the possibility of heat treating stainless steel foils for improving their ductility and malleability. This investigation revealed that the foils should be heated to 2000°F in a vacuum furnace then quickly quenched in water. The removal of the foil from the heat to a quenching tank must be instantaneous as the mass of the foil under treatment is very small.

Other factors considered were the effectiveness of heat treatment on relatively thin cross sections, proper crystalline alignment and the results of quenching shock. All local sources for possibly performing this operation were exhausted with the result that the test could not be undertaken.

(4) Physical Constants for Stainless Steel Foils.

Physical tests to determine Initial Modulus, Elongation and Tensile were conducted by an outside testing laboratory. The results of these tests are shown on Chart No. 2. All values are from an average of 6 samples tested of each type. Tests were performed on an Instron Tensilometer.

(5) Cleaning Stainless Steel Foils.

In the "as received" condition, stainless foils are coated microscopically with mill oils and other lubricating aids. Three approaches to cleaning were examined, (a) solvent, (b) heat cleaning and (c) acid cleaning.

A 1/2 hour immersion in acetone was employed for solvent cleaning following which the samples were dried in an air circulating oven.

The heat cleaning system involved exposure of the foils, unprotected, except for a metal envelope to circumvent flutter damage, in an oven atmosphere at 800°F for a period of 15 minutes. The resultant sample exhibited the typical bronze oxidation cast.

Acid cleaning was employed by immersion of the samples in a 35% hydrochloric acid solution for a period of 2 minutes, followed by a deionized water rinse and drying in a warm air circulating oven.

All samples were subjected to the water drop test. The solvent and acid cleaned foils exhibited little or no change in wettability, however, the heat cleaned foil showed a significant increase in wettability.

The stainless steel foil, grade 304, cold rolled .0001 supplied by the Hamilton Watch Co. of Lancaster, Penn., and cleaned by heat treatment at Dilectrix is a suitable material for application as a permeation barrier.

IV. Task II. Sample Preparation

A. Treatment of Foil.

Primarily the program was designed to investigate only foils which were tower coated with FEP and foils which had bee n primed with DuPont Teflon/aluminum primer (850-202) and then coated with FEP. Dilectrix' previous work with these systems had given indications that the bond strength attained would meet JPL's requirements of 13 lbs/in/in.

Based upon actual experience, where an inferior coating was obtained and pinholing of the foil occurred during the subsequent oven cycle, it became evident that the direct application of 850-202 primer coating was not feasible. Attempts at tower coating of the aforementioned primer also met with limited success.

As received, the 850-202 primer is extremely viscous and requires dilution. A normal mix was 1 part 850-202 primer to 3 parts deionized water (containing 7% by weight of Triton X-100). Pot life of this dispersion was approximately 10 minutes with a gradation of solids fall out starting approximately 3 minutes after mixing. Other formulations using 1 part 850-202 as a base and ranging from 1 part, 5 parts, 10 parts dilution of the same Triton/deionized water mixture resulted in too viscous a mix or repeatedly short pot life. Pick up of dispersion on aluminum foil in all cases was sparodic and non uniform.

This system was therefore discarded as a means of Teflon priming aluminum foils for the current contract.

The application of Teflon FEP in thicknesses of 1/4 mil and 1/2 mil to both surfaces of aluminum foil proceeded without incident.

Becuase of the difficulties encountered with the dip coating of DuPont's priming system, work began on a substitute chromate base priming system employing Alodine (Amchem Products, Inc., Ambler, Pa.). It was our intention to attempt to disperse Teflon/TFE into this mild acid etch solution and thereby formulate a workable primer, or one which would be readily adaptable to a dip coating or casting system.

One-half mil foil samples were then prepared using Alodine, Alodine/FEP dispersion, FEP dispersion only, and acidified FEP dispersions as the pre-treatment of aluminum foil, which were all heat treated for 30 minutes at 800° F.

The sample array are listed as follows:

A. TFE/FEP laminate film bonded to FEP coated foil.

B. Aluminum foil treated with Alodine solution and laminated to TFE/FEP film.

C. Aluminum foil treated with Alodine/FEP dispersion and laminated to TFE/FEP film.

D. Aluminum foil treated with FEP dispersion acidified with nitric acid, laminated to TFE/FEP film.

E. Aluminum foil treated with acidified (HCL) Alodine solution, laminated to TFE/FEP film.

F. Control - 2 sections of DF1700 film (FEP to FEP) bonded together.

Within the above groupings several variations of solids concentration and PH were explored.

Foil specimens, after treatment, were cut to 3-1/2" width x 4" long. Strips of DF-1700 were cut to 3-1/2" x 6" long. The laminate, therefore, consisting of a) DF-1700, b) Treated Foil, c) DF-1700, provided a 2" tab for peel tests. Laminated specimens were placed on a 5" diameter aluminum pipe, vacuum bagged and heat laminated following the same procedure employed in all other phases of this program.

This vacuum bag technique will be further discussed in a following section dealing with laminating parameters.

Results of this study on bonding systems as shown in Table III reveals that the bond strength of the control sample without foil exceeded any of the bonds of the foil/film laminates, and that the alodine foil treatment gave the greatest peel strength of the laminates.

PEEL TEST DATA
Table III

FOIL TREATMENT*	PEEL STRENGTH lbs/in.
None	0.33
FEP	2.75
Alodine	4.00
Alodine/FEP	2.58
FEP/Acidified (HNO_3)	1.50
FEP/Acidified (HCL)	0.66
Alodine/Acidified (HCL)	--
No Foil	5.37

* All Foils cured at 580°F/10 min. following treatment.

** 2 Strips DF-1700.

In an effort to ascertain the effect of cure temperature, additional tests were run on hand dipped samples of aluminum foils which were cured at temperatures ranging from 580°F to 680°F for 10 minute periods following the dispersion and/or solution treatment. The peel test data is given below in Table IV.

PEEL TEST DATA
Table IV.

FOIL TREATMENT	CURE TEMP.	PEEL STRENGTH lbs/In.
FEP	620°F	2.90
FEP	680°F	3.75
Alodine	580°F	4.92
Alodine/FEP	620°F	5.33
Alodine/FEP (2X)	680°F	5.75
Alodine/FEP (HCL)	680°F	5.75

These results confirmed the fact that elevated curing temperatures increased bond strength. A third series of tests was arranged. In this set aluminum foil samples which were tower coated with FEP were cut into smaller strips and cured at 680°F for 10 minutes. Control strips, which were not post cured, were tested also. The three lots of foil evaluated had been tower coated with variations in the FEP dispersion coating as follows:

437-1 1/2 mil foil; 1/4 mil FEP both sides

437-2 1/2 mil foil; 1/2 mil FEP both sides

437-3 1/4 mil foil; 1/4 mil FEP both sides.

Results of peel tests are given in Table V.

PEEL TEST DATA
Table V

CODE	FOIL TREATMENT	POST CURING TEMP	PEEL STR. lbs/in.
437-1 A	Tower Coated FEP	---	5.58
437-1 B	" " "	680°F	8.50*
437-2 A	" " "	---	2.08
437-2 B	" " "	680°F	4.00
437-3 A	" " "	---	2.25
437-3 B		680°F	1.66

* Film tab broke, maximum of 3 readings was 9.5 lbs/in. pull.

It is evident from the above data that tower coated foils, post heat treated, exhibit the highest bond strength. The data also supported Dilectrix' decision to use the 1/2 mil foil with the 1/4 mil FEP coating since this system of foil preparation resulted in highest bond values between base metal and film.

B. <u>Elimination of Blisters</u>.

Throughout the program, while work progressed on various priming systems, work simultaneously progressed on the elimination of blistering in the laminate structure. Blistering or gas pockets normally develop between the lower strata of film and the metal base. Microtome sections of the blistered areas in these film/foil composites confirm the relative location of the blisters or gas pockets. The elimination of this condition is important not only for the aesthetics of a smooth surface appearance but also for the necessity of having a continuation of the laminate bond between the substrate and the foil.

The factors leading to the presence of blistering may be explained in many ways and studies were conducted to prove or disprove each hypothesis.

for Teflon FEP was not adequate with regard to the removal or outgassing of the wetting agent in the FEP and, if this wetting agent was not thoroughly outgassed prior to aluminum foil lamination, it would outgas during subsequent Teflon FEP applications over the foil and would form blisters or delaminated areas at the substrate film/aluminum foil interface. To thoroughly explore this problem, all parameters were fixed and the temperature level for the outgassing cycle was fixed at 680°F. Twenty experimental test pipes were then constructed with varying time constant outgassing cycles, ranging from 30 minutes to 12 hours. The effects of outgassing (blistering) persisted in a random display. Since the time or dwell appeared to neither contribute or detract from this condition, this variable function was fixed. On all following sample preparation the thermal dwell was held at 1 hour at a temperature level of 680°F.

To further determine the effects of outgassing FEP coated foil the following was conducted. Two coated pipes were prepared by spray coating with 3 mils of TFE and .5 mils of FEP. Only one section was heat treated at 680°F for one hour. Three sections of foil were laid on each coated pipe. In each case the foil sections were dip coated with FEP and thermally pre-treated as follows:

Section 1 - No heat treatment

Section 2 - Treated for 10 minutes @ 680°F.

Section 3 - Treated for 60 minutes @ 680°F.

All of the foil sections on the pipe which were not heat treated developed some random blistering, while the sections of the foil on the pipe which was heat treated developed far less blisters in both size and frequency.

2b

It was apparent that the additional heat treatment of the substrate aided but did not completely eliminate totally the blistering conditions.

Upon the completion of the construction of the 20 sample sections as well as the heat treated base section mentioned above, it became increasingly apparent that the outgassing or blistering problem was inherent with the basic or substrate Teflon layering. It was determined therefore that additional effort in exploring this area was of primary importance. A program was arranged to explore the feasibility of creating an ultra porous base TFE film. The reasoning being that selective gas pockets forming under the metal foil would disperse evenly throughout such a structure thereby eliminating point blistering. The test series was initiated using two approaches. The first was to "dry spray" standard Teflon TFE dispersion onto the aluminum pipe substrate and the second was to formulate a totally new TFE dispersion using a finely divided TFE granular powder.

Pipe No. 091-737:

TFE-30 dispersion, dry sprayed onto an aluminum pipe mandrel with repetitive coating cycles prior to curing. A total build up of 3 mils was applied followed by a light coating of FEP and the part post heat treated for 1 hour at 680°F.

Pipe No. 091-738:

A dispersion or suspension of Teflon was formulated from finely ground TFE powder (particle sizes ranging from 5 to 10 microns). Thickening agents were added in order to hold the relatively large particles in suspension.

This material was spray applied to an aluminum pipe mandrel, sequentially coating, drying and curing. A thickness of 4 mils was applied followed by a light coating of FEP. The mandrel was then post heat treated for one (1) hour at 680°F.

In both cases, FEP coated aluminum foil was vacuum bag laminated to the base and additional FEP (3 mils) applied over the foil. While neither specimen exhibited any voids under the foil, the exceptionally rough surface of the base TFE apparently caused high point stresses in the metal foil resulting in numerous pinholes in the foil and causing abandonment of this approach.

To determine whether FEP contributed solely to the void entrapment problem a sample (091-739) was prepared eliminating TFE completely. The sample had a 3 mil FEP substrate under the foil and 3 mils of FEP over the foil. Surprisingly, no voids or gas pockets appeared anywhere in the specimen. When this specimen was twice reproduced (091-740 and 741) the necessity of an FEP interface between the TFE substrate and aluminum foil as a deterrent to void formation became apparent.

Samples (Figures II to V) submitted in Monthly Report No. 5 confirmed the fact that as the thickness of the FEP layer in the substrate between the TFE and foil increased from 0.25 to 1.0 mil, the blistering disappeared at about the 0.5 mil interface, as shown in Table VI following.

TABLE VI.

S/N	TFE	FEP	Al. Foil	FEP	Figures from Mthly. Rpt. #5	Appearance
091-702	3.0	0.25	437-1	3.0	II	Poor, numerous blisters
091-706	3.0	0.5	437-1	3.0	III	" "
091-708	3.0	0.5	437-1	3.0	IV	excellent, no blisters
091-710	3.0	1.0	437-1	3.0	V	" "

It is true that S/N 091-706 and S/N 091-708 appeared to be similar in construction, while one contained many blisters and the other none at all. However, a review of the fabrication data revealed the possibility of an error in measuring the FEP interface thickness in S/N 091-706.

The presence of voids or gas pockets which developed under the foil was now concluded to be the result of outgassing of TFE rather than FEP as had been suspected in the past. Voids were shown to be completely eliminated in an all FEP/aluminum foil laminate, and further eliminated in a laminate containing a TFE base where said base was coated with a heavier than normal FEP layer prior to foil lamination.

Another factor which influenced blistering in test samples, but which would not affect the aesthetics of bladder construction were the layers of Aqua-dag applied to the ends of the foil to facilitate bond or peel testing. The Aqua-dag was applied above the foil on one end and just below the foil on the other end of the pipe in order to obtain peel results at both interfaces. The presence of Aqua-dag (a colloidal graphite material employed as a release agent only) contributed to massive blistering only in those select "end" areas.

With the basic foil preparation techniques being standardized a final effort was made to change the vacuum bag laminating technique. As used the procedure initially was as follows:

A. Lay up foil onto Teflon coated mandrel.

B. Wrap the above arrangement with TFE slip sheet, bleeder fabric and place it in a TFE vacuum bag.

C. Place this assembly in a high pressure autoclave, equipped with heaters and controllers.

D. Maintain a vacuum of 29+ Hg on the bag and raise the pressure in the autoclave to 100 psig. Concurrently raise the temperature level to 580°F.

E. Maintain 580°F for a period of 5 minutes and then lower the temperature, drop the pressure and remove the assembly from the autoclave.

F. Remove vacuum bag, bleeder fabric and slip sheet and coat the aluminum/Teflon laminate with Teflon FEP.

G. Dissolve the mandrel substrate.

In the step by step process outlined above, probably the most important constants are temperature, vacuum and time. Since FEP has a distinct melt point at 540°F and continues to decrease in viscosity above this temperature level, this parameter was controlled carefully by means of thermocouples attached directly to the vacuum bag ports. Normally a temperature level of 580°F is maintained during final stages of vacuum laminating treatment. However, some consideration was given to the interdependence of the melt point of FEP (540°F) and the rate at which vacuum was applied to the confining TFE bag.

Four test pipes were run in the autoclave, S/N 091-713, 714, 717 and 755, in order to study this relationship. In each case the vacuum as measured in inches of Hg was held for a period of 30 minutes at the point where the pipe assemblies reached 540°F. Following this hold procedure, the normal vacuum of 29 in. of Hg was applied and the temperature raised to 580°F to complete the process. A summary of the test results are shown below in Table VII.

Table VII.

S/N	Treatment of Foil	Thickness	No. of Foil Sheets	In. of Hg @ 540°F in autoclave	Appearance
091-713	alodine/FEP	12 mils	2	1	very poor, large blisters
714	FEP	12 mils	2	2	excellent, small blisters on over-lap
717	alodine/FEP	7 mils	1	0	excellent; 2 small blisters
755	alodine/FEP	12 mils	1	2	very poor; large blisters

In comparing the above results with numerous previous runs on film/foil construction it is appropriate to conclude that the vacuum bag/autoclave process for laminating a treated aluminum foil to a Teflon substrate is adequate. The number of parameters and the variability of each, however, necessitate additional designing of controls and a deeper investigation of the effect of each of the constituents in the film/foil assembly.

C. Laminate Construction.

Dilectrix has previously shown that a 5% TFE co-dispersion (5% FEP/95% TFE) has exhibited improved physical properties over a TFE/FEP laminate. It was hoped that 10%, 30% or 50% co-dispersions might indicate an improved physical film structure while maintaining the normal appearance of a Teflon bladder film. Samples 091-742, 743, 744, 745, 747, 713 and 755 as listed below in Table VIII were fabricated for this purpose. The columns show the progression of each constituent as applied to the aluminum pipe substrate.

Table VIII.

S/N	TFE[1]	FEP[1]	Al. Foil Treatment	FEP[1]
091-742	3.0*	--	FEP	3.0
091-743	3.0*	0.5	FEP	3.0
091-747	3.0*	1.0	FEP	3.0
091-744	3.0**	0.5	FEP	3.0
091-745	3.0***	0.5	FEP	3.0
091-713	5.0****	1.5	alodine/FEP	6.0
091-755	5.0**	1.5	alodine/FEP	6.0

1) Thickness in mils
* 10% FEP used in co-dispersion with TFE.
** 30% " " " " " "
*** 50% " " " " " "
**** 5% " " " " " "

NOTE: Aluminum foil thickness in each case was 0.5 mils thick.

In order to obtain total fusion of the co-dispersion layer (substrate film) each spray application was sintered at a temperature of 680°F. This is appreciably above the 540°F melt point of the FEP.

Subsequently, the films containing the higher loading of FEP developed an uneven surface appearance. The unavoidable thermal degradation of FEP at this higher temperature contributed to this condition.

Samples 713 and 744 with 5% and 30% co-dispersion, respectively, both blistered. However, this was later proven to be the result of the foil treatment and not the TFE/FEP co-dispersion.

Also to obtain improved physicals, an attempt was made to prepare two aluminum foil laminates with TFE co-dispersion, instead of FEP, above the foil. Both of the laminates (S/N 091-749 and 749) were severly blistered upon application of the TFE top coat. This was expected prior to production, but it was felt that the low flex cycle life of the present laminate was not sufficient and an attempt should be made to upgrade this property. Due to the blistering, naturally this approach was not continued.

All of the scheduled laminate structures had thicknesses in the 7-8 mil range. However, as will be discussed the number of flex cycles required to pinhole the foil on the JPL flex tester was in all cases less than ten (10)

Laminate thickness was thought to be the problem area. Three (3) twelve (12) mil laminate structures (S/N 091-713, 714, 755) were therefore fabricated with a basic construction of 5.0 mil TFE co-dispersion, 1.5 mil FEP, .5 mil Al. foil (treated with FEP) and 6.0 mils FEP. Flex cycle life before pinholing of the al. foil did not improve.

However, these thicker laminates all had initial moduli in the range of 250,000 psi, as compared to the 400,000 psi with the thinner structures. This appears to be one advantage of heavier laminate structures.

V. Task III. Testing and Results.

A. Permeation.

The basic Vango Permeation Cell was designed by JPL and is shown in Figure 1 of Jet Propulsion Laboratory Technical Memorandum No. 33-55. Six permeation cells were reworked to incorporate a stainless steel permeant reservoir and sample holder in place of the costly and breakable glass apparatus. The results of permeation testing has shown all aluminum foil laminates to have zero permeability over the 24 hour test period. Even with small pinholes in the foil, permeation was not evident as long as the Teflon film itself was not destroyed. Apparatus employed in permeation testing is shown as Exhibit 4 in the Appendix.

B. Bond Test.

Another of the important considerations of this study was the bond strength at the film/foil interface. The bond strength peel tests were set up on selective pipe structures by introducing a thin layer of Aqua-dag at the interface layers, directly under and over the foils, at both ends of the pipe assembly. The pull tabs created by this procedure were cut into 1" wide strips and peeled apart on a Scott Tensilometer at a jaw separation rate of 2"/min. with the pull tabs 180° apart.

Surprisingly, the alodine treated foil laminates had a slightly lower bond strength than the FEP coated foil. Also, the thicker laminates had a slightly lower bond strength than the thinner ones. All of these peel results are lower than the ones shown in Section IV on foil treatment and is apparently due to the size of the bonding area.

All of the test samples and other selected samples, were exposed to N_2O_4 for a 96 hour period. In every case delamination of the foil was evident. This was largely due to the all-around edge exposure that these samples received in the test apparatus. Bladders would not be subjected to this exposure and consequently would not show the propensity to delaminate that these 1" x 3" samples did.

Attached in Appendix 5 is one sample of S/N 091-714 before and after exposure to N_2O_4. Shown below in Table IX is a summary of bonding results.

TABLE IX..

PULL TEST BEFORE N_2O_4 SOAK.

S/N	Bond Strength (#/in/in)	Thickness of Laminate	Foil Coating
091-705	3.5	7 mil	1/4 mil FEP both sides
091-711	2.7	7 mil	1/4 mil FEP both sides
091-714	2.2	14 mil	1/4 mil FEP both sides
091-717	2.5	7 mil	alodine treat; 1/4 mil FEP both sides
091-713	2.2	14 mil	" " " " "
091-755	1.6	14 mil	" " " " "

AFTER N_2O_4 SOAK TEST (96 hour)

S/N	Appearance After Soak - 2 Samples
091-705	Delaminated completely on one side; other side delaminated at edges and easily peeled.
091-708	Delaminated completely on both side.
091-710	Delaminated completely on one side; other side delaminated at edges and easily peeled.
091-711	Delaminated completely on both sides.
091-741	One delaminated completely on both sides. One same as S/N 091-705, only harder to peel.
091-743	One delaminated completely on one side; other side delaminated at edges and easily peeled. One delaminated completely on both sides.
091-744	Delaminated completely on one side; other side delaminated at edges and easily peeled.
091-747	Delaminated completely on one side; other side delaminated at edges and hard to peel.
091-714	Delaminated completely on both sides.
091-717	Delaminated completely on both sides.
091-713	Delaminated completely on one side; other side delaminated at edges and hard to peel.
091-755	Delaminated completely on one side; other side delaminated at edges and hard to peel.

C. Flex Test.

Another area which has met with limited success throughout this program is the flex life of the film/foil composite laminates. As shown in Exhibit 7 the JPL flex tester (rolling fold simulator) was too rigorous a test for these laminates. The different elongations of the foil (approximately 40%) and Teflon (approximately 400%) caused pinholing of the foil in less than 10 cycles in all cases; in a few samples pinholes occurred as soon as the laminate passed over the end Vee section. The test was performed at 70°F, with 5 lb. tension on the sample.

To further explore the effect of heavier film structures in this type of test, three (12-14 mil) laminates (S/N 091-713, 714, 755) were fabricated. S/N 091-714 is shown as Exhibit 6 after ten (10) flex cycles and, as can be observed, pinholes are evident in the foil. Although the foil exhibits low flex life, the adjacent Teflon film will endure normal flex fatigue. For example, flex failure occurs after several thousand flexes.

D. Surface Appearance.

Included in the following table (Table X) is a summary of the construction of all samples with a blister free surface appearance. All of the pipes included either did not blister, or if there was a slight blistering it could safely be presumed to be due to lack of proper or adequate FEP in the sub-layer directly under the metal foil.

TABLE X

PIPES WITH ACCEPTABLE SURFACE APPEARANCE

S/N	APPEARANCE	CONSTRUCTION From Pipe Up Thickness in Mils
091-705	Fair-blisters near Aqua-dag at edges; few towards center	5% co-dispersion TFE (3.0): FEP (0.5): Foil* Aqua-dag: FEP (3.0)
091-711	" " " " " " "	5% co-dispersion TFE (3.0): FEP (1.0): Foil Aqua-dag: FEP (3.0)
091-708	Excellent - none	5% co-dispersion TFE (3.0): FEP (0.5): Foil: FEP (3.0)
091-710	" "	5% co-dispersion TFE (3.0): FEP (1.0): Foil: FEP (3.0)
091-739	" "	FEP (3.0): Foil: FEP (3.0)
091-740	" "	" " " " "
091-741	" "	" " " " "
091-743	" "	10% co-dispersion TFE (3.0): FEP (0.5): Foil: FEP (3.0)
091-744	Very good - two small blisters	30% co-dispersion TFE (3.0): FEP (0.5): Foil: FEP (3.0)
091-747	Excellent - one contaminated area with large blisters	10% co-dispersion TFE (3.0): FEP (1.0): Foil: FEP (3.0)
091-714	Very good - very small blisters on overlap of two foil layers	5% co-dispersion TFE (5.0): FEP (1.5): Foil: Aqua-dag: FEP (6.0)
091-717	Very good - two small blisters	5% co-dispersion TFE (3.0): FEP (1.0): Foil: Aqua-dag: FEP (3.0)

*NOTE: In all cases except 091-717 the foil is FEP coated only. S/N 091-717 used alodine/FEP coated foil.

E. Physical Constants.

For convenience stress-strain data was broken down into two tables. Table XI is a complete compilation of the physical constants of all of the pipes produced under this contract, while Table XII summarizes the properties for those constructions which produced blister free laminates.

The data has shown the foil laminates to have the physical properties which were expected from work conducted prior to this program. The tensile stress of the samples ranged from 2500 to 2700 at the yield point. The corresponding elongation at the yield point was from 23 to 41%. It should be noted that the higher elongations, S/N 708, 710, 711, were from those samples which had no greater than 1 mil FEP layer in the substrate and were made with a 5% TFE codispersion. These same samples were the ones which generally exhibited the higher tensile (3150-3500) and elongation 430% at the break point of the film. Other results being equal, this appears to be the type construction which offers the highest mechanical properties. Low initial moduli, however, are a characteristic of thicker laminates which have also been shown to possess maximum tensile and yield values.

TABLE XI

S/N	Thickness (Mils)	Laminate Preparation	Appearance
Order of Production		*TFE is 5% co-dispersion unless noted by A=10%, K=30%, L=50% Foil is 437-1 (1/2 mil foil with 1/4 mil FEP) except as noted in 713, 717 & 755 A.D. is Aqua-dag.	
701	8.25	TFE 3.0:Foil:AD:FEP 3.0	Poor (lg.blisters)
702	7.46	TFE 3.0:Foil:FEP 3.0	Poor (long blisters)
703	8.73	TFE 3.0:Foil:AD:FEP 3.0	Fair (slight nr, center excessive - edge.
705	9.10	TFE 3.0:FEP.5:Foil:AD FEP 3.0	Fair ("as 703")
706	8.57	TFE 3.0:FEP.5:Foil:FEP 3.0	Poor (excessive medium blisters)
707	9.22	TFE 3.0:FEP.5:Foil:AD FEP 3.0	Fair (good at center lg.blisters at edges)
708	8.56	TFE 3.0:FEP.5:foil:FEP 3.0	Excellent
709	9.20	TFE 3.0:FEP 1.0:Foil:AD FEP 3.0	Fair ("as 707")
710	8.63	TFE 3.0:FEP 1.0:Foil:FEP 3.0	Excellent
711	9.53	TFE 3.0:FEP 1.0:Foil:AD FEP 3.0	Fair ("as 703")
727-B	7.49	TFE 3.0:FEP.5:Foil:AD FEP 3.0	Very good (some medium blisters)
731-C	7.79	TFE 3.0:Foil:AD FEP 3.0	Excellent
732	7.62	TFE 3.0:FEP .5:Foil:FEP 3.0	Poor (many large blisters)
737-A	11.21	TFE 4.0 (Dry spray disp.) FEP.5:Foil :FEP 3.0	Rough coating

TABLE XI contd.

S/N	Thickness (Mils)	Laminate Preparation	Appearance
738	9.92	TFE 5.0(wet spray disp.):FEP .5:Foil:FEP 3.0	Rough Coating
739	7.87	FEP 3.0:Foil:AD:FEP 3.0	Excellent
740	8.08	FEP 3.0:Foil:FEP 3.0	Excellent
741	7.91	FEP 3.0:Foil:FEP 3.0	Excellent
742	7.46	TFE(A) 3.0:Foil:FEP 3.0	Fair (some blistering)
743	8.85	TFE(A) 3.0:FEP.5:Foil: FEP 3.0	Excellent
744	9.06	TFE(K) 3.0:FEP.5:Foil: FEP 3.0	Very good (couple small blisters)
745	9.53	TFE(L) 3.0:FEP.5:Foil: FEP 3.0	Poor (blisters throughout)
748	9.26	TFE(K) 3.0:FEP.5:Foil: FEP.5:TFE(K) 3.0	Poor (blisters throughout)
747	10.04	TFE(A) 3.0:FEP 1.0:Foil FEP 3.0	Excel.(one area with large blisters)
749	8.69	TFE(K) 30%: FEP 0.5:Foil: 3.0 TFE(K) 30%	Poor (blisters throughout)
713	14.38	TFE 5.0:FEP 1.5: Alodine/ FEP Foil:AD:FEP 6.0	Poor (blisters throughout)
714	14.05	TFE 5.0:FEP 1.5: Foil:AD: FEP 6.0	Very good (small blisters on overlap of two foil layers)
717	8.00	TFE 3.0:FEP 1.0:Alodine/ FEP Foil: AD:FEP 3.0	Very good (2 small blisters)
755	14.63	TFE(K) 5.0:FEP 1.5:Alodine/ FEP Foil:AD:FEP 6.0	Poor (blisters throughout)

TABLE XI contd.

S/N	Strain Rate (% min.)	Modulus Kpsi 4000	8000 **	% Elongation Alum.	Film	Tensile (Psi) Yield	Max.
	100	395.1	399.5				
701	10	503.6	482.1	39.0	357	2772	3035
	100	357.1	374.5				
702	10	361.6	397.1	24.0	422.0	2649	3429
	100	356.6	378.8				
703	10	449.2	471.2	36.5	430	2700	3353
	100	343.3	360.3				
705	10	460.4	428.1	35.1	445	2695	3304
	100	303.9	318.1				
706	10	337.5	363.5	23.0	476.0	2582	3632
	100	336.7	368.7				
707	10	389.9	430.7	38.7	436	2710	3230
	100	369.5	383.9				
708	10	400.7	399.6	40.0	439.0	2707	3489
	100	356.7	376.3				
709	10	485.8	477.2	38.8	452	2678	3293
	100	323.8	343.7				
710	10	420.4	430.9	41.0	437.0	2702	3448
	100	337.7	341.3				
711	10	400.0	401.3	39.4	429	2641	3151
	100	346.4	379.1	yield	break		
727-B	40	430.7	449.3	386.0	532.0	3220	2212
	100	341.7	360.7				
731-C				277.0	546.0	2984	2385
	100	328.2	330.8				
732				345.0	413.0	3103	1871
	100	256.0	235.0				
737-A				31.6		2000	1928
	100	64.4	64.7				
738				7.8	42.7	1457	1325
	100	356.3	380.7				
739	10	392.1	452.1	23.9	510.	2830	3674

**10,000 where thickness is greater than 11 mils.

TABLE XI contd.

S/N	Strain Rate (% min.)	Modulus Kpsi 4000	Modulus Kpsi 8000	% Elongation Alum.	% Elongation Film	Tensile (Psi) Yield	Tensile (Psi) Max.
	100	307.5	337.3				
740	10	402.1	390.3	31.6	67.0	2735	2482
	100	364.0	383.2				
741	10	455.5	464.3	26.5	41.0	2763	2470
	100	370.7	391.4				
742	10	509.2	502.7	39.5	324.0	2682	3076
	100	350.5	372.7				
743	10	431.7	443.4	38.0	400.0	2654	3133
	100	321.4	346.3				
744	10	435.4	424.7	33.6	288.0	2634	2687
	100	337.3	364.1				
745	10	411.4	412.4	18.2	32.6	2514	2282
	100	268.3	280.1				
748	10	299.1	293.6	19.2	410	2432	3054
	100	324.4	333.1				
747	10	406.5	399.2	37.4	374.0	2574	2906
	100	279.8	293.6				
749	10	289.5	290.9	12.8	320	2382	2703
	100	242.8	244.6				
713	10	261.0	262.8	37.0	438	2450	3248
	100	216.3	224.3				
714	10	255.8	255.1	40.8	469	2485	3346
	100	335.9	369.2				
717	10	428.6	413.5	35.2	397	2821	3357
	100	230.3	241.5				
755	10	274.7	264.3	33.6	437	2485	3003

TABLE XII

STRESS/STRAIN DATA (Ranked by Tensile at Yield Point) For Specimens With Smooth Surfaces:

S/N	8000 psi Initial Modulus 10%/min Strain Rate kpsi	8000 psi Initial Modulus 100%/min Strain Rate kpsi	Yield Point Tensile (psi)	Yield Point Elongation (%)	Maximum Tensile (psi)	Maximum Elongation (%)
739	452.1	380.7	2830	23.9	3674	510
717	413.5	369.2	2821	35.2	3357	397
741	464.3	383.2	2763	26.5	2470	41 (delam)
740	390.3	337.3	2735	31.6	2482	67 (delam)
708	399.6	383.9	2707	40.0	3489	439
710	430.9	343.7	2702	41.0	3448	437
705	428.1	360.3	2695	35.1	3304	445
743	443.4	372.7	2654	38.0	3133	400
711	401.3	341.3	2641	39.4	3151	429
744	424.7	346.3	2634	33.6	2687	288
[illegible]7	399.2	333.1	2574	37.4	2906	374
714	255.1	224.3	2485	40.8	3346	469

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VI. Conclusions.

The work performed under JPL Contract No. 952091, Phase II, has been most constructive in solving some of the problems heretofore associated with the assembly and construction of a metallic foil Teflon film bladder structure. All of the work accomplished within the scope of this contract was necessarily performed on standard cylindrical pipe bases and as such may not be truly correlatable to full scale bladder assemblies.

The problem areas that have existed in the past, however, were more clearly brought to light and solutions were developed. Perhaps the most perplexing problem has been the persistance of gas pockets or blisters in the film/foil composite and the particular stage in processing at which these blisters begin to occur. A logical sequence of planned experiments followed within the contract has led to a satisfactory solution to this problem.

Achievement may be listed as follows:

A. A selective choise of aluminum foil and fixed thickness gauge has been determined.

B. Several methods of surface cleaning and priming of the metal foil were explored in detail. Although the priming system resulted in a measureable degree of bond of foil to film. this area requires additional exploration.

C. Independent of the priming system and Teflon film thickness under and over the metal foil, gas entrapment or blistering has been eliminated.

D. The vacuum/temperature/pressure process for laminating the metallic foil to a Teflon base has been sequentially arranged and controlled so that a high degree of reproducibility has been achieved. The effects, however, of large segments of metal foil and frequency of overlaps at foil edges on composite integrity did not fall within the purview of this contract and do require further study.

Due to the nature of some of the processing problems which developed during the exercise of this contract, some deviations were necessary resulting in the fabrication of many more sample pipe structures than were originally anticipated. Since the inclusion of specimens from each of the composite samples within this report would be impractical, a selection of sample structures was made and are appended to this report in Appendix C.

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VIII. APPENDIX

Exhibit 1 - Pinhole Frequency Photo #1

Exhibit 2 - Pinhole Frequency Photo #2

Exhibit 3 - Pinhole Frequency Photo #3

Exhibit 4 - Vango Permeability Set-up

Exhibit 7 - Rolling Fold Simulator

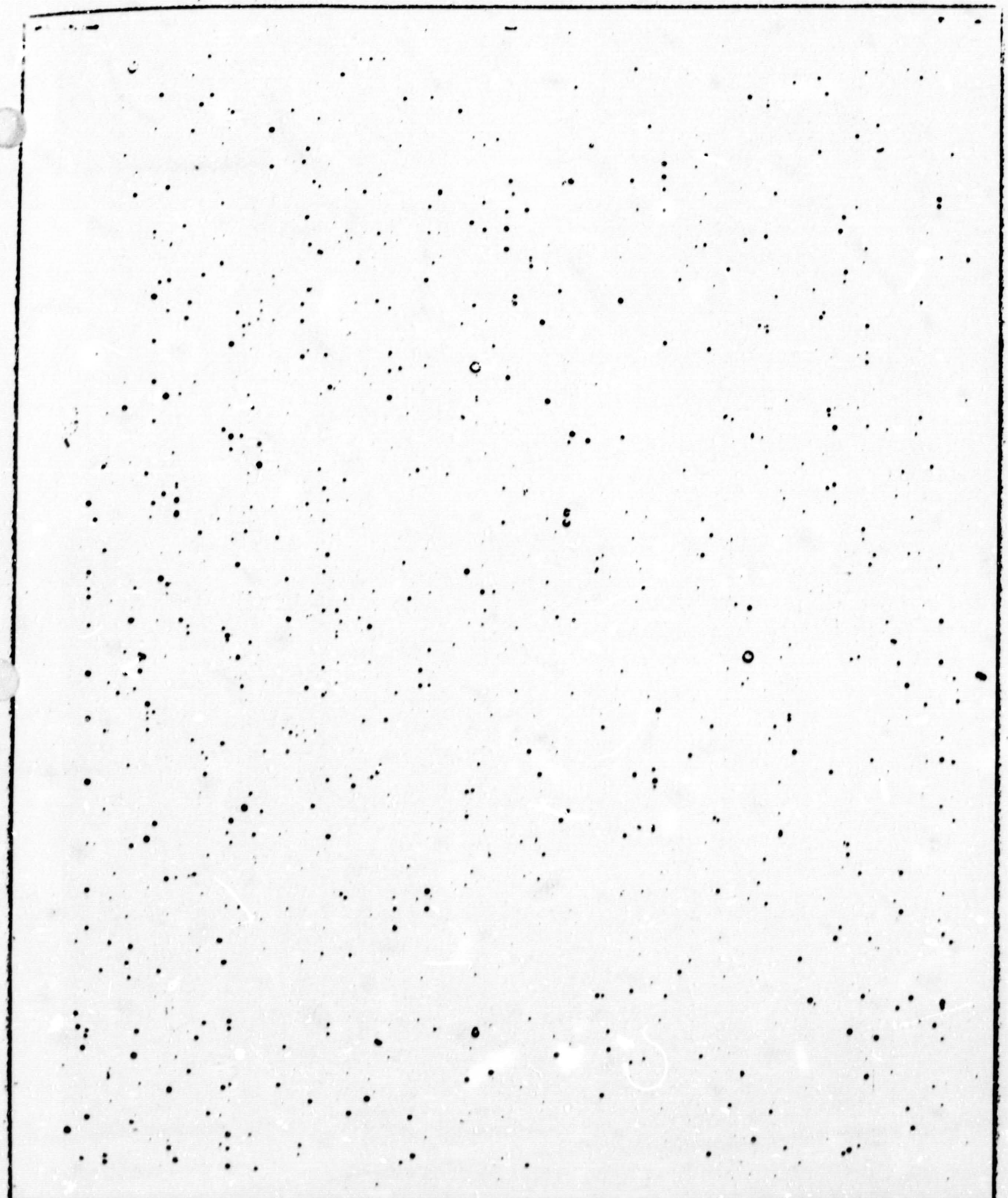

Exhibit 1
Pinhole Frequency Photograph
Republic Foil Corporation
.00025 Electrodry Aluminum Foil
Photo #1 Case #16895 Roll #1

Exhibit 2

Pinhole Frequency Photograph
Republic Foil Corporation
.00025 Electrodry Aluminum Foil
Photo #2 Case #16895 Roll #2

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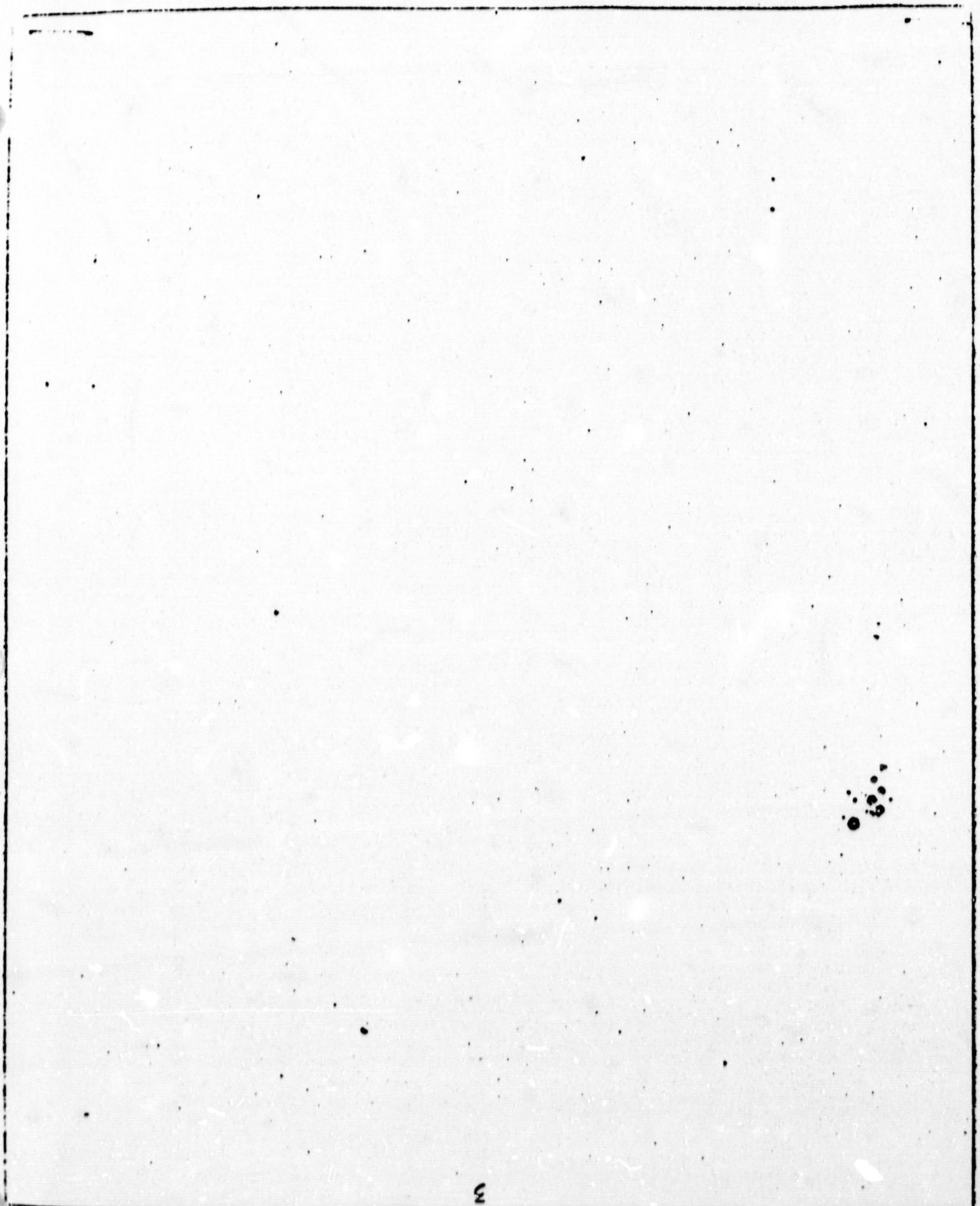

Exhibit 3
Pinhole Frequency Photograph
Republic Foil Corporation
.0005-1 Side Bright Aluminum Foil
Photo #3 Case 781

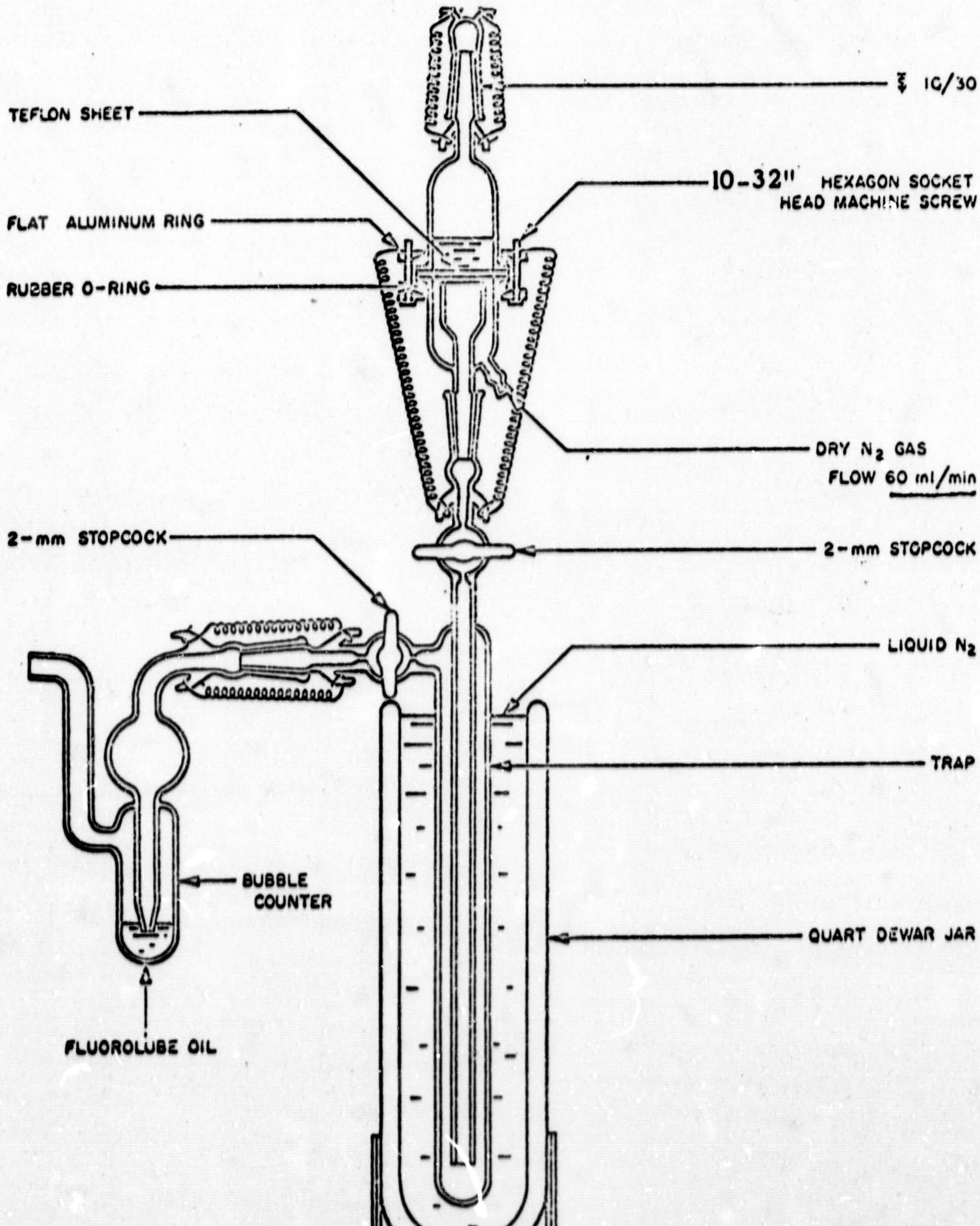

Exhibit 4
Vango Permeability Setup.

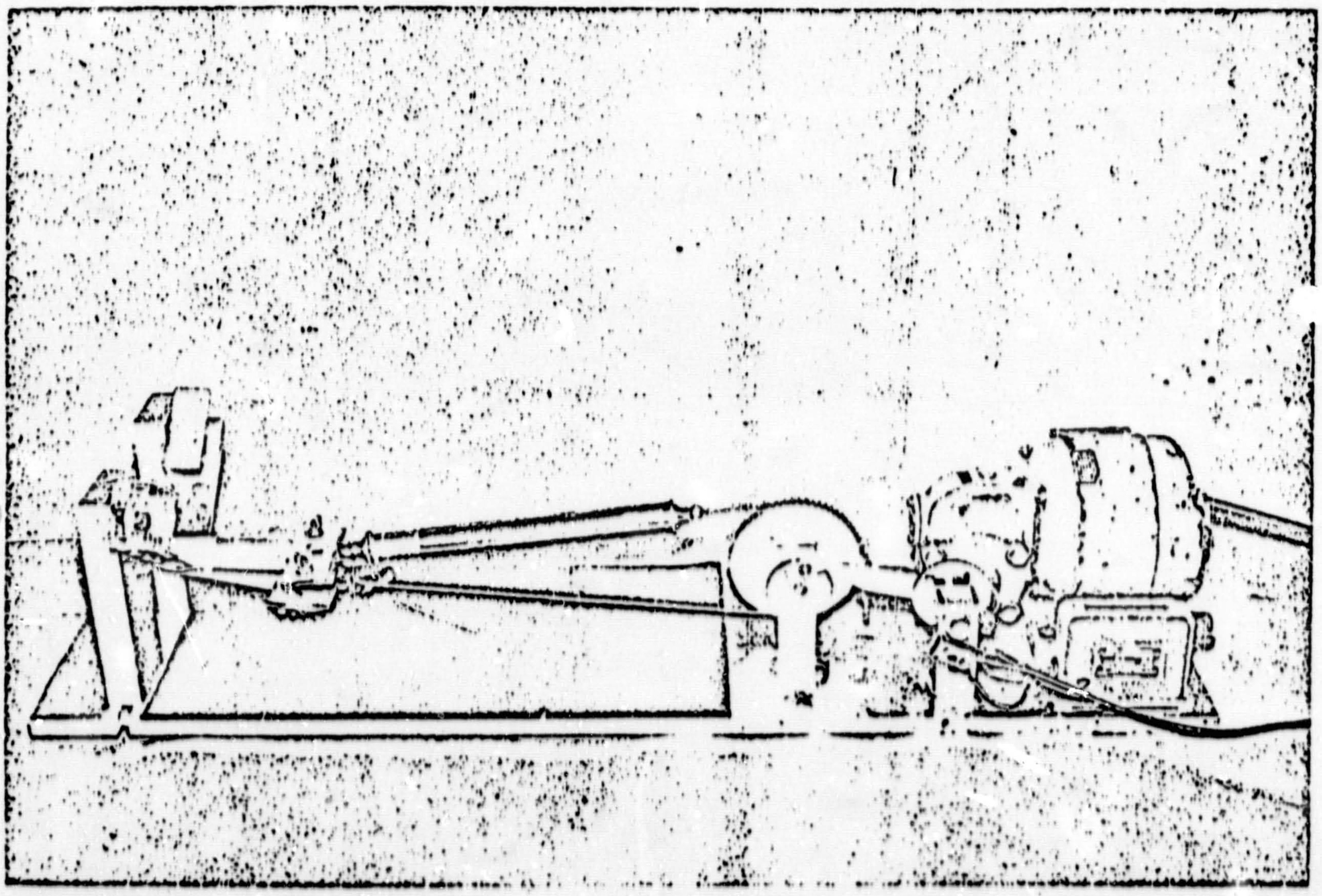

Exhibit 7. Rolling Fold Cimulator